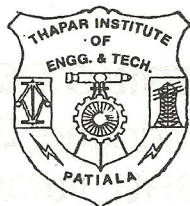


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DEVELOPMENT OF SiO_2 COATINGS ON METALS BY SOL-GEL TECHNIQUE

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ABSTRACT

SiO_2 coatings on different metal substrates like aluminium, stainless steel etc were prepared by sol-gel dip coating technique. Tetra Ethyl Ortho Silicate (TEOS) solution (40%) in ethyl alcohol was used as the precursor for silica coatings. Hydrolysis of TEOS was effected with water to obtain coating solutions of suitable consistency for coatings. Various metal substrates were mechanically polished and chemically etched in suitable pickling solution to activate the metal surface for coating and then dip coated in already prepared silica coating solutions. The substrates were withdrawn at a constant rate of 2mm / sec. The coatings were subjected to heat treatment at different temperatures. The thickness of the coatings was measured and the surface of the coated specimen was characterised by scanning electron microscopy. The corrosion resistance of the coatings was measured in 3.5% NaCl solution and also in 5% H_2SO_4 acid solution by the standard potentiodynamic polarization technique. It was observed that the corrosion resistance of the SiO_2 coated Al metal is about 65-92% as compared to the bare Al metal. Silica coating on stainless steel was of nano-sized, with a pleasant light golden colour. The above coatings have high commercial value particularly for corrosion and abrasion resistant applications.

INTRODUCTION

Ceramic oxide coatings are generally applied on metal surfaces due to their resistance to corrosion, high temperature oxidation [1-2], thermal barrier, abrasion resistance, dielectric [3-4], optical, and other functional properties. For example, amorphous silica is deposited on aluminium and stainless steel substrates for corrosion resistance applications. Due to their chemical and thermal durability, SiO_2 coatings are of interest for protection of metals against acid corrosion and oxidation. Generally, coatings are applied on metal substrates by sol-gel technique because metal substrates of complex geometry can be coated at low temperature by this simple and low cost process [5-6]. The process is mainly based on the hydrolysis and condensation reaction of organometallic precursors in alcoholic solution [7-13].

In the present work, silica coatings were prepared on commercial grade aluminium metal as well as on stainless steel substrates by dip coating technique. The aluminium metals are also coated by deposition of silica vapors generated due to decomposition of TEOS. The corrosion resistance was measured by potentiodynamic polarization technique using 3.5% NaCl and 5% H_2SO_4 solutions and the results were compared with respect to bare Al metal.

EXPERIMENTAL PROCEDURES

Metal substrates of area (5cm x 1cm) were used for silica coatings. The substrates were polished by sand paper and washed thoroughly, dipped in a standard pickling solution and then ultrasonically cleaned. The coating solutions were prepared using 40% tetra ethyl ortho silicate (TEOS) hydrolysed with appropriate amount of water and alcohol to produce coating solution of suitable coating consistency. The metal strips were dipped into the coating solution vertically and were withdrawn at a constant rate of about 1mm/sec. Similarly, silica coatings were also prepared by deposition of silica vapors produced by the decomposition of concentrated TEOS. The substrates were exposed in the path of silica vapors such that the metal surface is coated uniformly. The coatings were dried at 80°C for 10 hrs. Finally silica coated aluminium and stainless steel substrates were heat-treated at temperature of 400°C and 650°C respectively for 1 hr.

CHARACTERIZATION

The thickness of the coated specimen was measured by an electronic instrument, which works on the principle of eddy current. The amorphous nature of the coating was confirmed by XRD technique. The surface morphology of both dip coated and vapor deposited silica coatings on aluminium metal were characterized by a scanning electron microscope (SEM) and are presented in fig. 1 and fig. 2 respectively. The corrosion measurement was also carried out by potentiodynamic polarization technique. Typical polarization curves of bare and silica coated aluminium substrates of different coating thickness are presented in fig. 3. The typical surface morphology of silica coatings on stainless steel substrate characterized by scanning electron microscope (SEM) is also presented in fig. 4. Silica coatings were uniform through out the surface of stainless steel with a pleasant light golden colour. The particle sizes are in the form of nano range.

MEASUREMENT OF CORROSION RESISTANCE

Corrosion resistance of the coated and uncoated specimens was evaluated by electrochemical technique, utilizing a standard potentiodynamic polarization unit. The polarization unit can record current (I) - potential (E) curves with a sweep rate of 0.6 mV/sec. From the I-E curves, the potential Vs log i curves were generated on a semi-log plot. The linear portion of the tafel region i.e. between 60 mV to 120 mV of over potential (η) is extrapolated to the open circuit potential (OCP) value of the corroding metal to get the corrosion current density (i_{corr}). From this i_{corr} value, the corrosion rate in mills per year (mpy) was calculated by using the equation derived from Faraday's laws of electrolysis.

$$R \text{ (mpy)} = 0.129 a i_{corr} / nD$$

Where R is the corrosion rate in mills per year, (mpy)

a is the atomic weight of the corroding metal

i_{corr} is the current density in $\mu\text{A}/\text{cm}^2$

n is the valence of the corroding metal

RESULTS

The measured corrosion rate in terms of mills per year (mpy) along with corrosion

current density (i_{corr}), corrosion potential (E_{corr}) of both dip coated and vapor deposited silica on aluminium substrates are presented in table 1 and table 2 respectively.

Table 1. Corrosion current density (i_{corr}), corrosion potential (E_{corr}) and corrosion rates (mpy) measured from the polarization curves for dip-coated silica

Corroding medium	Thickness (μm)	i_{corr} ($\mu\text{A}/\text{cm}^2$)	E_{corr} (mV)	Corrosion Rate (mpy)
5% H_2SO_4 solution	0	35.0	-590	15.05
	5	10.0	-560	4.30
	16	9.0	-530	3.87
3.5% NaCl solution	0	14.0	-750	6.02
	5	12.0	-750	5.16
	16	4.6	-740	1.978

Table 2. Corrosion current density (i_{corr}), corrosion potential (E_{corr}) and corrosion rates (mpy) measured from the polarization curves for vapor deposited silica.

Corroding Medium	Thickness (μm)	i_{corr} ($\mu\text{A}/\text{cm}^2$)	E_{corr} (mV)	Corrosion Rate (mpy)
5% H_2SO_4 solution	0	35.0	-590	15.05
	5	4.3	-590	1.849
3.5% NaCl solution	0	14.0	-750	6.02
	5	2.7	-750	1.161

DISCUSSION

From table 1, the corrosion current density (i_{corr}) of bare aluminium metal is 35 mA/cm² and corrosion rate is 15.05 mpy in 5% sulphuric acid medium. In case of dip coated silica coatings, the corrosion rate sharply reduces to 4.3 mpy for a coating thickness of 5 mm which was further reduced to 3.87 mpy with a coating thickness of 16 mm. Therefore,

the corrosion rate was reduced by 74% compared to bare metal.

Similarly, the i_{corr} value of bare metal tested in 3.5% NaCl solution is 14 mA/cm² and the corrosion rate was about 6.02 mpy. By applying silica coatings of 5mm thickness the corrosion rate decreased marginally to 5.16 mpy, which is about 14% less compared to bare aluminium metal. To reduce the corrosion rate appreciably, thick coatings of nearly 15mm are required which reduce the corrosion rate about 67% compared to bare metal.

Corrosion resistance of silica coatings by vapor deposition technique is excellent in both corrosive solution. From table 2 it is observed that the corrosion rate of bare aluminium metal in 5% H₂SO₄ solution is 15.05 mpy. By applying 5mm of silica coating the corrosion rate was sharply reduced to 1.849 mpy, which is 90 % less compared to bare metal.

Similarly the corrosion rate of bare aluminium metal in 3.5% NaCl solution is 6.02 mpy. By applying 5mm of silica coating the corrosion rate was sharply reduced to 1.161 mpy, which is about 80% less compared to bare metal. This is attributed to the fine particles of silica vapor deposited densely over aluminium surface covering the entire surface of Al metal, furthermore there is no loss during heat treatment due to absence of organic components and water molecules. Corrosion resistance of vapor deposited samples are more than the dip coated samples because of the uniform coatings generated by the vapors covering almost all the exposed area of the substrate.

CONCLUSIONS

From the above study, the following conclusions are drawn.

1. Silica coatings from the TEOS were successfully coated on Al metal by dip coating and vapor deposition technique. From the potentiodynamic polarization study, it was found that the silica coatings could resist the corrosion rate by 65-90% compared to the bare Al metal.
2. The corrosion rate sharply decreases with the application of 5mm silica coatings in case of vapor deposition technique. In case of dip coated silica the decrease in corrosion rate is somewhat less due to the presence of some organic components, which affects the coatings during heat treatment process.

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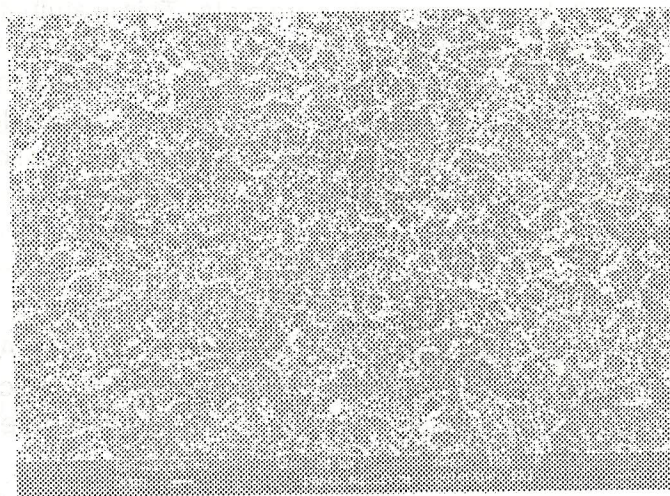


Fig.1: Surface morphology of dip coated silica on aluminium metal substrate

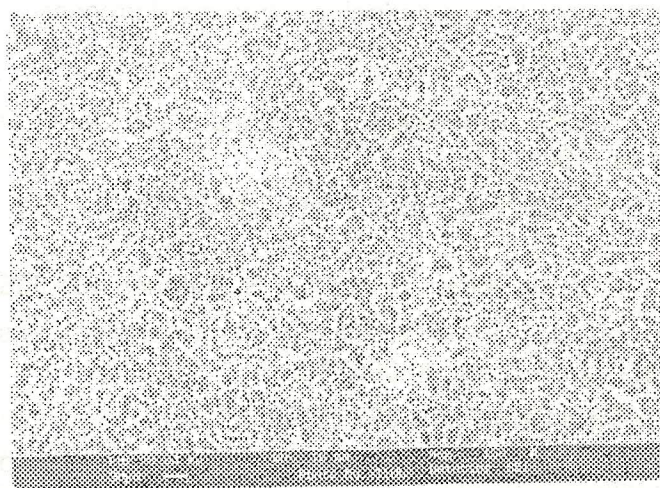


Fig.2: Surface morphology of vapor deposited silica on aluminium metal substrate

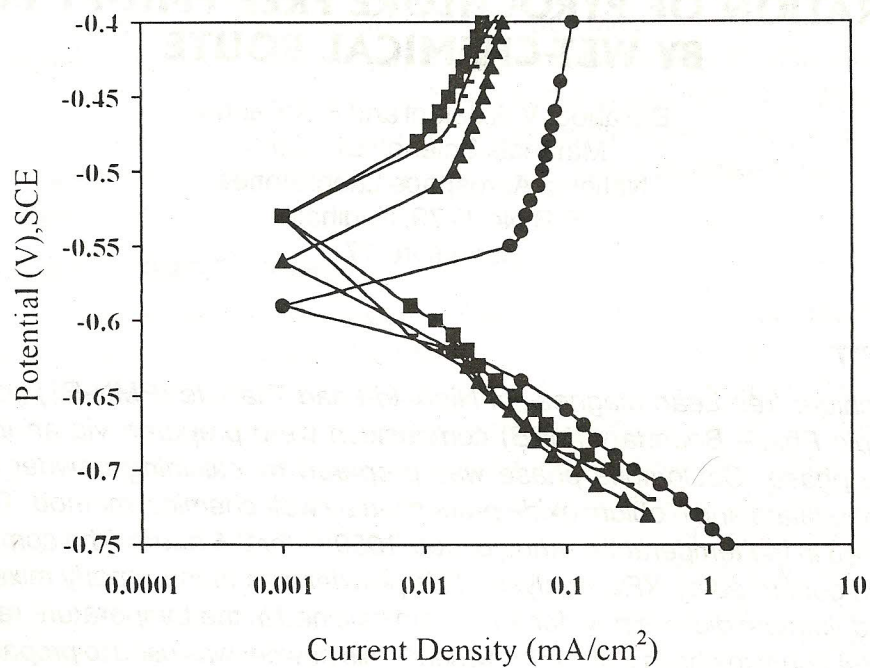


Fig.3. Typical polarization curves of (•) Al metal, and silica coatings on Al metal of thickness (σ) 5mm, (v) 12mm, (–) 16mm prepared by hydrolysis of TEOS in 5% H_2SO_4 medium.

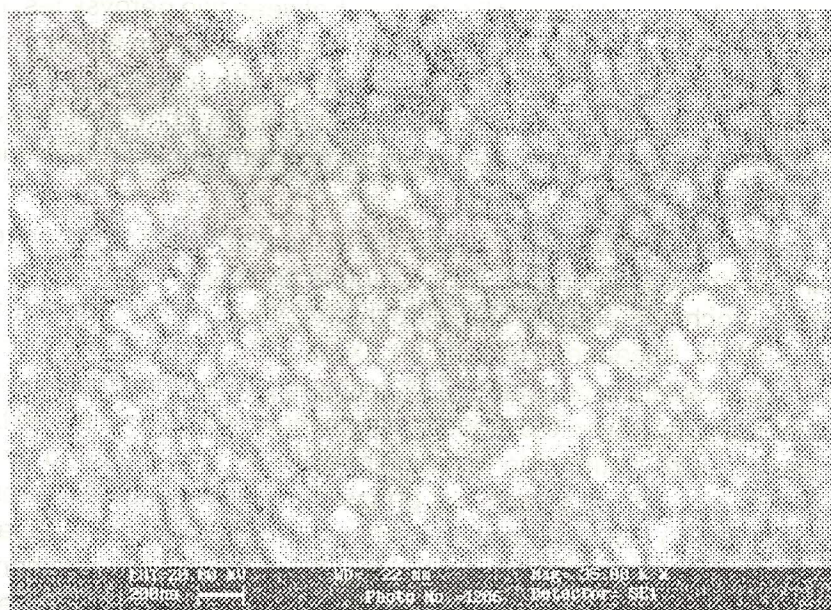


Fig.4: Typical surface morphology of silica coatings on stainless steel substrate